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DILATOMETRIC BEHAVIOUR OF A COLD-BONDED CERAMIC

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DILATOMETRIC BEHAVIOUR OF A COLD-BONDED CERAMIC

by

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ABSTRACT

The dilatometric behaviour of an experimental cold-bonded material produced by Relmech Manufacturing Ltd., Elmira, Ontario during heating in air at approximately 10°C/min to its melting point has been determined using a hot-stage projection microscope.

From an analysis of the projected image of the progressively heated sample recorded on film, the material was found to exhibit a slight shrinkage of about 1.5% to 1000°C followed by a sharper expansion up to a maximum of 4% reached at close to 1250°C. Thereafter, the material rapidly collapsed and was completely melted at 1280°C.

Keywords: Bonded Ceramics, Dilatometry, Microscopy.

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COMPORTEMENT DILATOMÉTRIQUE D'UNE CÉRAMIQUE AGGLOMÉRÉE À FROID

par

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RÉSUMÉ

Une analyse du comportement dilatométrique d'un matériau expérimental aggloméré à froid, produit par Relmech Manufacturing Ltd., Elmira (Ontario) a été effectuée. Le matériau a été chauffé à environ 10°C/min jusqu'à son point de fusion et un microscope à projection à platine chaude a été utilisé pour déterminer son comportement.

L'image de l'échantillon qui avait été chauffé progressivement a été enregistrée sur film et analysée. L'analyse a révélé que le matériau démontrait un léger retrait, soit environ 1,5 %, à 1 000°C. Ce retrait a été suivit d'une dilatation rapide, jusqu'à un maximum de 4 %, à une température de près de 1 250°C, après quoi, le matériau s'est rapidement affaissé et a complètement fondu, à 1 280°C.

Mots-clé : Céramiques agglomérées, Dilatométrie, Microscopie.

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INTRODUCTION

In part, the physical characteristics of a material can be defined by its dilatometric behaviour and the progressive {in the case of multi-phase materials} or sudden {in the case of a pure compound} development of liquidus on heating. Both stages, expansion-contraction and melting as a function of temperature, are not only characteristic of a material; they are also important in applications where the product is to be used at elevated temperatures where distortion, creep with time, and other changes are critical. These and other changes such as grain growth, sintering kinetics and phase development can be readily investigated by means of a heating microscope.

In the present case, only the expansion-contraction and melting behaviour were required and consequently a small representative sample was examined using a commercial hot-stage projection microscope.

PROCEDURE

Sample Preparation

A representative sample in the form of a small cube with dimensions of about 3x3x3 mm was cut from a "dog bone" test bar using a diamond saw. The material was of unknown inorganic chemical composition but known to contain the polyaramide: kevlar. The sample was only identified as RML-2AF: treated Kevlar, 30% NaOH, Temp. 40-45°C, Time 47 h.

Sample Examination

The progressive shrinkage of the material with increasing temperature was determined using a Leitz Heating Microscope, type IIAP, which is capable of operating in air up to 1750°C. The overall configuration of this instrument is shown in Fig. 1.

In essence, the microscope consists of three components: a tungsten filament light source {left-hand component of Fig.1} that passes light through the furnace assembly {central unit} to a monoocular microscope equiped with long focal length lenses that allow observation at up to 40x {right-hand unit}.

The sample is placed on the flattened end section of a thin horizontal alumina tube containing a 6/30 Pt-Rh thermocouple which reaches to the centre of the Pt-40%Rh-wound electric furnace. The thermocouple bead is located approximately 2 mm below the sample and is connected directly to an analogue string galvanometer to give a continuous readout of temperature. In actual operation, two images are viewed in the microscope: the silhouette of the sample viewed on a frosted 24x36 mm groundglass grid with the temperature reading of the galvanometer placed immediately below.

During heating in a static air atmosphere at 9°C/min, the sample silhouette and temperature were recorded on 35-mm film at nominal 100°C intervals up to the fusion or melting point of the material using an attached Leica M 4 camera. Up to approximately 1000°C, the luminance of the light source was greater than that of the sample and hence it is seen in silhouette. However, above that temperature the luminance of the sample dominates, hence the light source was reduced to half power to gain adequate contrast between

the image and the background. In this manner, a series of photographs were obtained showing the progressive shrinkage and deformation of the sample as shown in Fig. 2.

Treatment of Data

A qualitative examination of Fig. 2 shows that the material first shrinks slightly to about 1000°C then expands to about 1240-1250°C and finally melts at 1280°C. In order to quantify these changes, each image was first enlarged, the area of each determined in arbitrary units using an XY tablet of a Zeiss Videoplan system and finally the change in the square root of the area with respect to the original room-temperature value was plotted versus temperature.

The Videoplan is a powerful image analysis system that is capable of receiving input from an XY tablet using photographs, projected images as in the present case or any video image that can be viewed directly on the system monitor and features edited by the operator using the tablet and stylus. The software allows a wide range of image features to be quantitatively analysed such as length, area, perimeter, feret's diameter, major- and minor-axes, etc, together with a complete range of stereological and statistical analyses of the chosen parameters.

For convenience, each 35-mm negative image was projected directly onto an XY tablet using an enlargement of sevenfold and the outline digitized directly into memory by tracing with a pen stylus. The option of determining the area of the enclosed figure traced by the stylus using an automatic close feature was selected from the software menu. Five separate determinations of the same outline showed that the standard deviation of the area was less than 0.3% of the mean. Because the sample silhouette is approximately square, the area was related to a linear dimension by taking the square root of the projected area and using these values to determine the percentage length change that occurs on heating. Table 1 gives the area of each projected enlarged image, the length and percentage length change determined in this manner.

The data on expansion and contraction of the sample versus temperature are plotted in Fig. 3. The size of the square points used to plot these data is approximately 10 times the standard deviation of the length data. It can be seen that the material suffers little dimensional change up to 400°C. Above that temperature, a slow general shrinkage occurs with imposed expansions at approximately 700°C and 900°C possibly as a result of phase changes. The overall shrinkage to 1000°C, which is presumably due to dehydration of various phases present, amounts to approximately 0.7%.

The expansion in the temperature range 1000 to 1260°C may be due either to the progressive formation of a liquid phase in the sample or the development of a phase having a higher specific volume. Extensive liquid phase was developed at 1270°C as indicated by the rounding of the specimen, Fig.2. With further temperature increase complete melting occurs at about 1280°C.

CONCLUSIONS

Material RML-2AF displays dimensional stability up to about 400°C, thereafter a progressive shrinkage amounting to ~1.5% at 1000°C occurs. Between 1000 and 1240°C, the sample expands to 4% at 1240°C. Complete melting occurs at ~1280°C.

Table 1. Variation of Sample Geometry with Temperature

Temperature °C	Projected Area[1]	Length[2]	% Length Change[3]
RT	4536	67.4	0
250	4535	67.3	-0.01
500	4464	66.8	-0.80
600	4440	66.6	-1.07
700	4595	67.8	0.65
800	4437	66.6	-1.10
900	4531	67.3	-0.06
1000	4471	66.9	-0.73
1110	4728	69.2	2.79
1180	4938	68.8	2.09
1200	4811	70.3	4.34
1220	4894	69.4	2.98
1240	4502	70.0	3.88
1260	4371	67.1	-0.37
1270	3694	66.1	-1.83
1278	3021	60.8	-9.76
1280	MELTED	-	-

(1) Given in arbitrary units
(2) Taken to be the square root of (f1)
(3) Normallized with respect to the initial length at RT

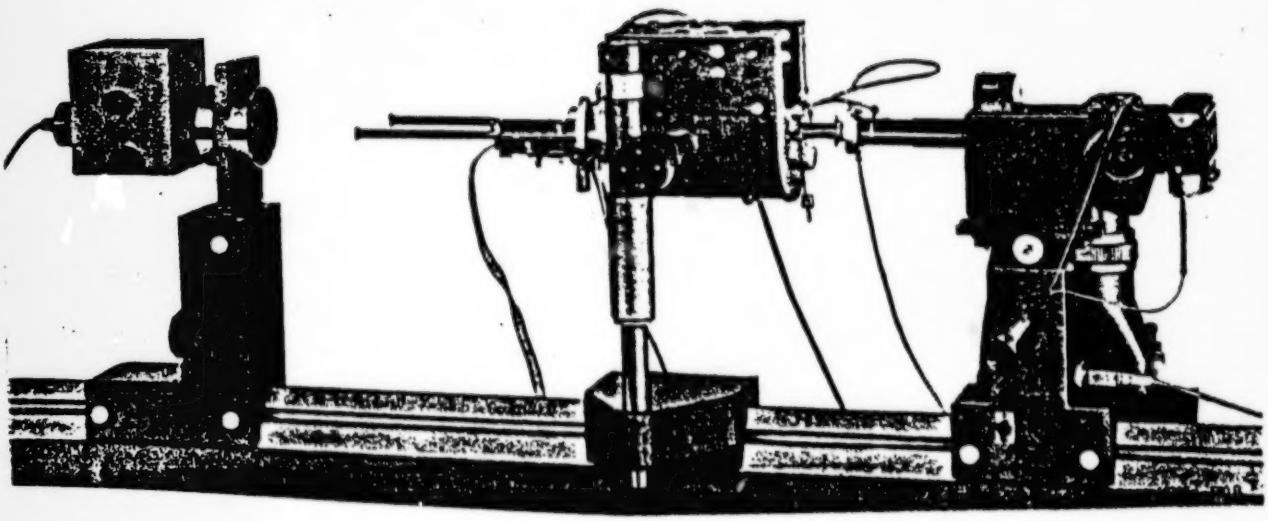

Fig. 1. General view of Leitz heating microscope showing at the left-hand side the light source, in the centre the 1750°C furnace and at the right-hand side the photomicroscope with built in 35-mm camera and string galvanometer.

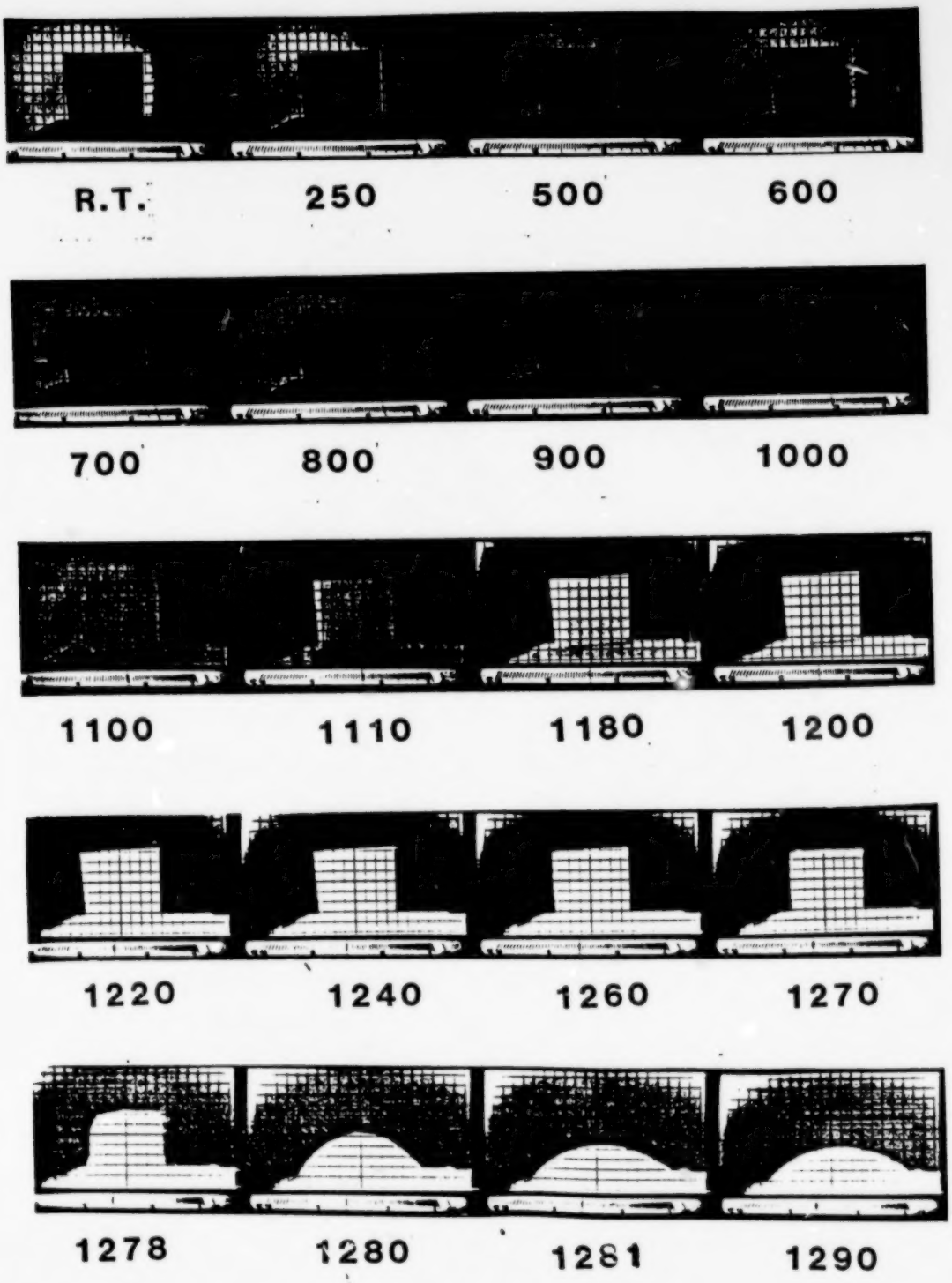

Fig. 2. View of RML-2AF sample in heating microscope during heating in air at approximately 9°C/min to the temperatures indicated.

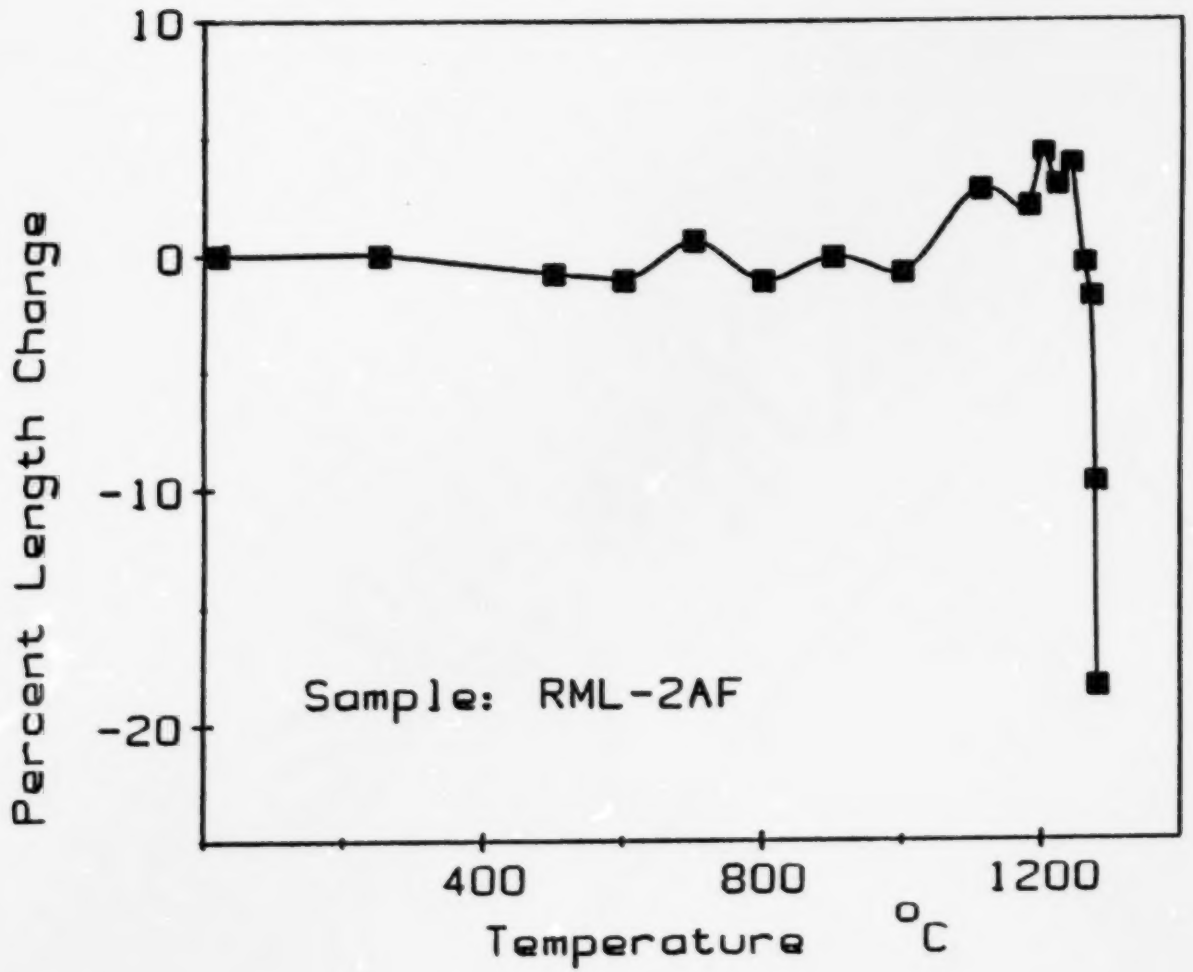

Fig. 3 Variation of sample length with temperature derived from data given in Table 1.